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Translation of "Spechennyi mednyi poroshok." Tsvetnyye metally, no. 7, 1960, pp. 72-77.

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An aluminum-based heat-resistant material, specifically, a sintered aluminum powder known as SAP, has been lately the object of widespread study. This material is a compound of pure aluminum with aluminum oxide, and it is prepared by the powder metallurgy method. Very fine powders are usually used in SAP preparation. This compound is similar in structure to that of a highly resistant heat-treated aluminum alloy, but without the tendency of the cementing particles to coagulate on heating. Such SAP properties as thermal and electrical conductivity, resistance to thermal shock, and capability of distorting in the hot state are of particular interest. SAP possesses exceptionally high strength characteristics at high temperatures. However, the yield strength, and also the long-time and fatigue strengths, of this SAP material are considerably exceeded in the 300° to 500° C range by those of heat-treated aluminum alloys (1, 2, 4).

The SAP alloy's high heat resistance has incited investigators to start studying other oxide-strengthened powder metallurgical compounds. In particular, Grant, Preston, and others carried out investigations of copper and nickel properties with dispersions of various sized silica and alumina particles. The data obtained by the authors show that oxides added to copper increase the latter's heat resistance and raise the recrystallization temperature (3, 5).

Data concerning the copper alloy's long-time strength at higher temperatures show that these alloys are characterized by a flat pattern in the long-time strength curve, which is an indication of the structure's thermal stability. By comparing the value of the long-time strength for 1000 hours, we find that the copper alloy with 10 percent Al_2O_3 at 350° C is four times as strong as pure copper, and at 450° C, it is three times as strong.

In the work by Grant and Zwilsky, it is noted that the most effective admixture appears to be the aluminum oxide (5). Similar data concerning the iron - aluminum oxide composition were recently published (6). In this way, not only SAP but other metal-oxide compounds appear to be stable at high temperatures (in relation to high temperatures).

*"Spechennyi mednyi poroshok." Tsvetnyye metally, no. 7, 1960, pp. 72-77.

The structure, the electrical resistance, and the mechanical properties of a sintered copper powder (SCP)[1] with an alumina, silica, or magnesia admixture are investigated in the present paper. Powders used as raw materials for those investigations are described in table I.

METHOD OF SCP SAMPLE PREPARATION

To obtain SCP, mixtures were prepared by simple mechanical mixing of copper powder and oxides in appropriate proportions. This mixing took place in steel ball mills by the wet method. The ratio of balls to the charge was 5 to 1. The time of mixing was 48 hours. The operation had to be carefully carried out, since the distribution and uniformity of the dispersed phase and, consequently, the properties of the finished product depend upon the efficiency of the mixing process. Mixtures were prepared from copper powders with 1, 3, and 5 percent (in terms of volume) of both alumina and silica and with 1, 3, 5, and 10 percent (in terms of volume) of magnesia.

The prepared mixtures were subjected to hydrogen reduction at 350° C for 30 minutes. From the reduced mixtures, cakes 80 mm in diameter and 110 to 120 mm in height were prepared by hydrostatic compression. The compression pressure was 1000 kg/cm^2. These cakes were sintered in the hydrogen atmosphere with a slow temperature rise to 1000° C and maintained at this temperature for 3 hours. The sintered preparations, 70 mm in diameter, were extruded at 800° C into bars 21 mm in diameter, which then were tempered at 400° C for 1 hour. Further investigations were carried out on these pressed and sintered samples.

MICROSTRUCTURES OF THE SAMPLES

The samples prepared by the method described above were subjected to metallographic investigation. Figure 1 presents photomicrographs of the samples with 1 percent Al_2O_3. The clear areas represent the copper alloy foundation, the dark ones the oxide phase. In the samples with 1 percent oxide, the basic copper grain dimension is 3 to 5 μ, and that of the oxides up to 1 μ.

The photograph shows that the aluminum oxide is not only distributed along copper grain boundaries but also within the grains.

As may be seen from figure 2, all the samples have densities close to their theoretical values.

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[1]In the following SCP will mean sintered copper powder.

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SCP samples with 1, 3, and 5 percent SiO_2 constitute an exception, as their densities are, respectively, 96, 95.5, and 96 percent of the theoretical density.

ELECTRICAL CONDUCTIVITY OF THE SCP COMPOUND

The electrical conductivity was measured by the compensation method over samples of 6-mm diameter and 35-mm length. It may be seen from table II that the SCP electrical conductivity decreases as the oxide content percentage increases. A particularly sharp drop in conductivity is observed in SCP compounds with 5 percent SiO_2 or Al_2O_3. In spite of the lowered electrical conductivity, relative values for SCP with 1 to 3 percent Al_2O_3 still remain high and are within the limits of 87 to 93 percent of the conductivity of pure copper.

TEMPERATURE OF SCP RECRYSTALLIZATION

The study of recrystallization was conducted by Vickers hardness measurement of sintered samples after annealing at different temperatures for 1 hour. The annealing took place at 200°, 300°, 350°, 400°, 500°, 600°, 700°, 800°, and 900° C. Figure 2 shows the hardness as a function of annealing temperature (as a matter of comparison, data for both cast and sintered pure copper are also shown). The curves of the SCP sample hardness have a flat course in comparison with the pure copper, which is explained by the increase in copper recrystallization temperature and by the absence of coagulation of hardening oxide particles. The hardness of the SCP with 1 percent Al_2O_3 at room temperature is close to that of the pressed pure copper. As the annealing temperature is raised to 300° C, recrystallization begins in the pure copper and the hardness falls rapidly, while in the SCP with 1 percent Al_2O_3 no recrystallization takes place until much higher temperatures, as indicated by the flat hardness curve. In the SCP with 5 percent Al_2O_3 a significant increase in hardness at room temperature is observed in comparison with the pure copper.

With the annealing temperature increased to 600° C, the hardness of the SCP with 5 percent Al_2O_3 remains practically unchanged, but above 600° C a more significant hardness decline is observed. The SCP sample with 3 percent Al_2O_3 occupies an intermediate position, also keeping a flat curve up to high annealing temperatures. It must be pointed out that, in spite of the decline of hardness after high-temperature annealing, it still remains high for SCP with 3 to 5 percent Al_2O_3, the respective hardnesses being 130 and 153 kg/mm^2 after annealing at 700° C for 1 hour.

It may be seen from figure 3 that the SCP hardness increases with magnesium oxide content and reaches 182 kg/mm^2 for SCP with 10 percent MgO at 20° C temperatures. With the increase of annealing temperature, a slow decline in hardness may be observed. The same may be said of SCP samples with 1, 3, and 5 percent SiO_2. These curves differ little from the preceding. The flatness of the curves is also preserved, as in the case of SCP with Al_2O_3 and MgO (until temperatures of 700° to 800° C are reached).

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When comparing the influence of the various oxides on SCP hardness dependence on temperature, it may be noted (figs. 2 and 3) that SCP samples with 1 percent alumina, magnesia, and silica have a nearly similar hardness along the full extent of the curve. The hardness curve of SCP with 3 percent alumina lies considerably higher than the corresponding curves with magnesia and silica, and it is even higher than those for 5 percent magnesia or silica. In this way, SCP samples with 3 to 5 percent Al_2O_3 have higher hardness values at all annealing temperatures in comparison with the corresponding SCP samples with magnesia and silica. The experiments establish that SCP recrystallization temperature lies 300° to 400° C higher than that of the pure copper.

TENSILE STRENGTH OF THE SCP ALLOY

In all experiments on stress-rupture, all the SCP alloy samples prepared with oxides were 5 mm in diameter and 25 mm in length. The experiments were carried out on samples which were cut from the bars extruded at 800° C and then subsequently annealed at 400° C for 1 hour. For comparison, samples of cast and powder-metallurgy pure copper were prepared and tested under the same conditions as the SCP samples. In comparing the tensile strength of the SCP with various oxides, a certain difference in the absolute tensile-strength values in relation to the percentage of oxide content and to the temperature of the test is apparent. The curves of figure 4 show the tensile strength of the SCP with Al_2O_3 content under conditions of room and high temperature, and the same may be said of SCP alloys with MgO and SiO_2.

The improvement of the tensile strengths with the increase of oxide content is particularly noticeable at room temperature and at 400° C. At higher test temperatures (600° and 800° C) tensile strength also rises with increasing oxide content, but less sharply than at room temperature. The lesser tensile strength isotherm slope, characterizing a weaker influence of the oxide admixtures at higher temperatures, may be explained by a rising plasticity of the SCP copper matrix. In comparing the tensile-strength values of the corresponding SCP samples with various oxides, it may be noted that at 20° and 400° C the strength of SCP with Al_2O_3 differs little from that of SCP with MgO and SiO_2. However, with the increase of test temperatures to 600° to 800° C, the tensile-strength values of SCP with Al_2O_3 and MgO are higher than those of SCP with SiO_2. So, the tensile strength of SCP with 3 to 5 percent Al_2O_3 in tests ranging from room temperature to 800° C is correspondingly decreasing from 27 and 33 to 5.9 and 6.3 kg/mm^2, while it correspondingly falls from 28 and 30 to 3.6 and 3.7 kg/mm^2 for SCP with 3 to 5 percent SiO_2.

Therefore, the tensile strength of oxide-added SCP samples considerably surpasses that of pure copper and other SCP alloys at temperatures ranging from room temperature to 800° C.

THE SCP SCALING RESISTANCE

The scaling resistance of samples of SCP with 1 percent alumina, magnesia, or silica, and, for the sake of comparison, pure copper was determined. The sample dimensions were: diameter, 15 mm; height, 8 mm. The oxidation was carried out at 500°, 600°, and 700° C, holding for 1 hour. Figure 5 presents data of the weight increase of copper and other samples in relation to the oxidation temperature. As the diagram shows, the SCP weight-increase curves are located below that for pure copper, which suggests a lower oxidation of SCP alloys with oxides. The absolute values for SCP with various oxides differ little from one another.

CONCLUSIONS

1. A technology for obtaining samples and products from a sintered copper powder (SCP) with alumina, magnesia, or silica has been worked out, and their properties have been investigated.

2. The strength of oxide-added SCP considerably exceeds that of copper alone at temperatures from room temperature to 800° C. Similarly, the strength of the Al_2O_3-added SCP at testing temperatures of 20° to 800° C exceeds the strength of copper by $1\frac{1}{2}$ times. As to the SCP hardness, it is two to three times that of copper.

3. The oxide admixture to copper increases its recrystallization temperature from 300° to 600° or 700° C. The SCP does not lose its hardness after heating to the above mentioned temperatures.

4. The electrical conductivity of SCP, with 1 to 3 volume percent of Al_2O_3 is equal to 87 to 93 percent of the conductivity of copper.

5. The SCP scaling resistance is higher than that of copper.

6. SCP shows best results with the Al_2O_3 admixture, and, in that case, the values of strength and hardness are higher when applying a high-dispersion oxide and uniformly distributing it throughout the product's volume.

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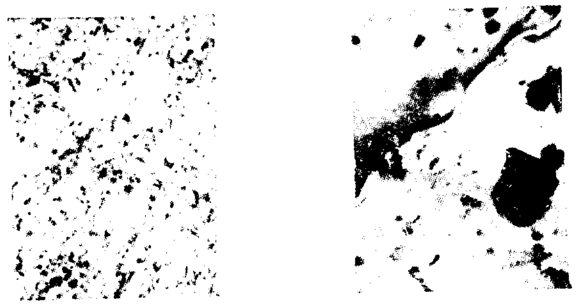

(a) X1500. (b) Photograph by electron microscope. X8800.

Figure 1. - SCP microstructure with 1 percent Al_2O_3.

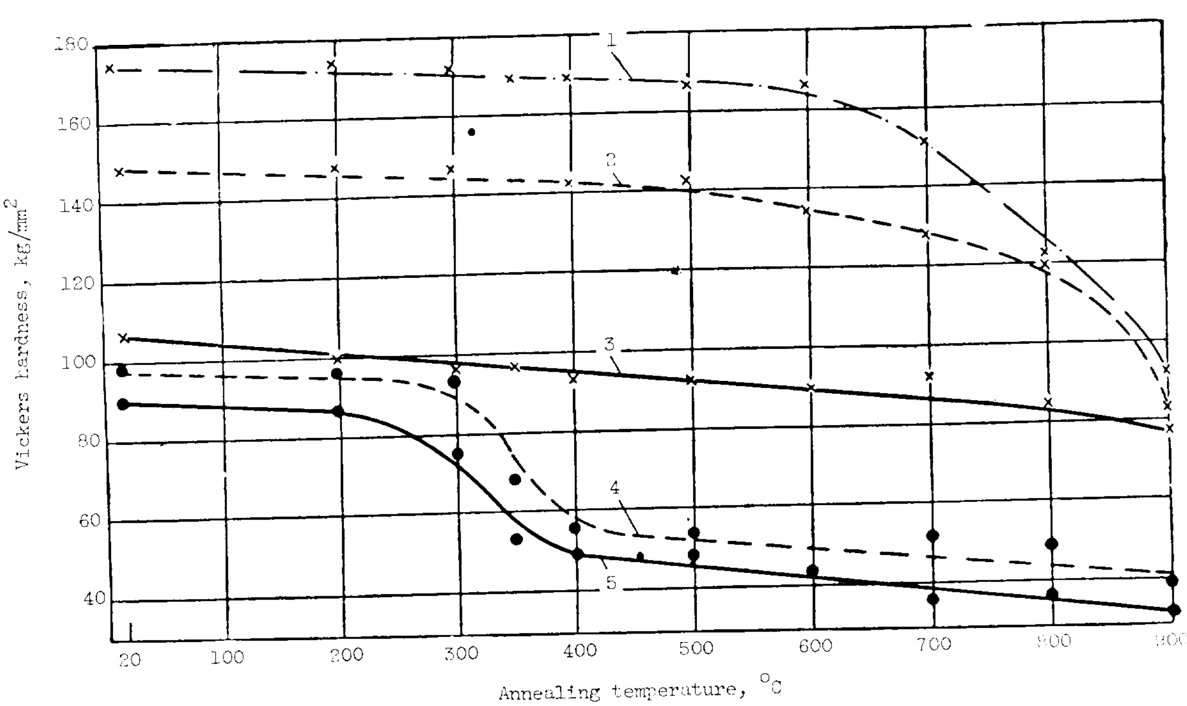

Figure 2. - Dependence of hardness upon annealing temperature for copper and SCP with Al_2O_3. 1, SCP plus 5 percent Al_2O_3; 2, SCP plus 3 percent Al_2O_3; 3, SCP plus 1 percent Al_2O_3; 4, copper powder; 5, cast copper.

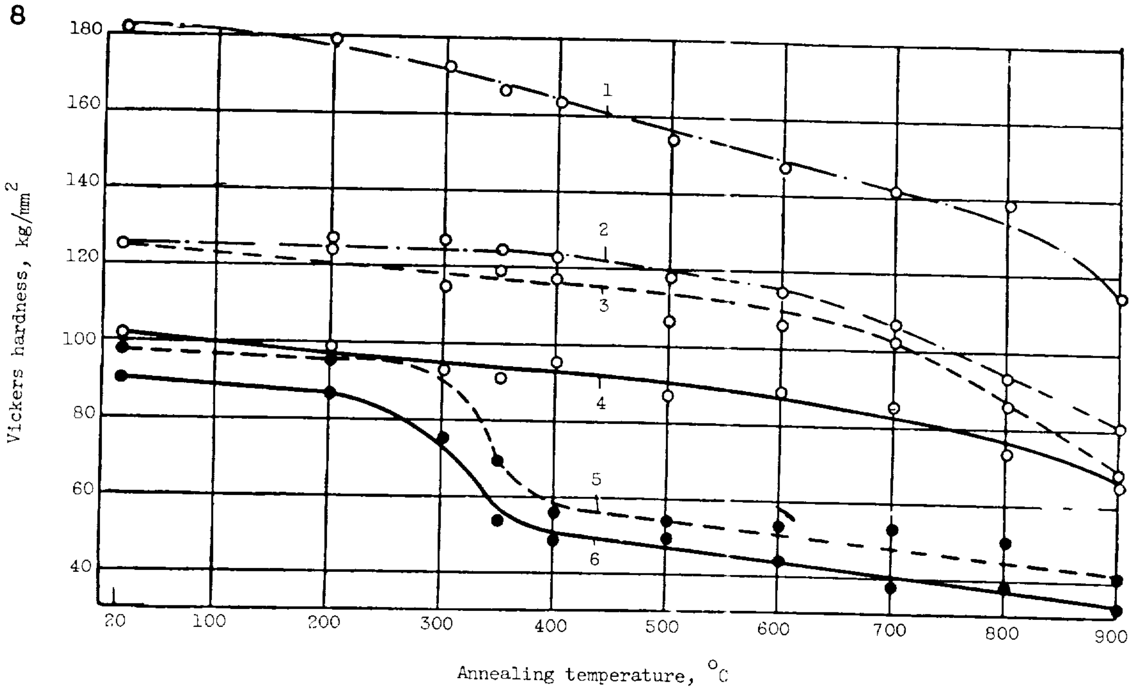

Figure 3. - Dependence of hardness upon annealing temperature for copper and SCP with MgO. 1, SCP plus 10 percent MgO; 2, SCP plus 5 percent MgO; 3, SCP plus 3 percent MgO; 4, SCP plus 1 percent MgO; 5, copper powder; 6, cast copper.

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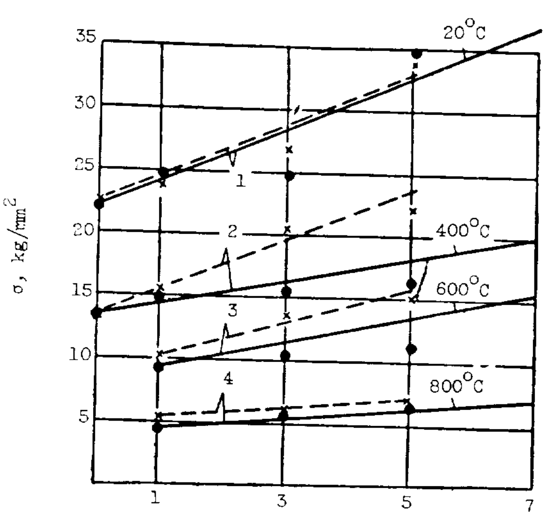

Figure 4. - Dependence of tensile strength on percentage of Al_2O_3 (dashed lines) and MgO (solid lines) at different temperatures. 1, 20° C; 2, 400° C; 3, 600° C; 4, 800° C.

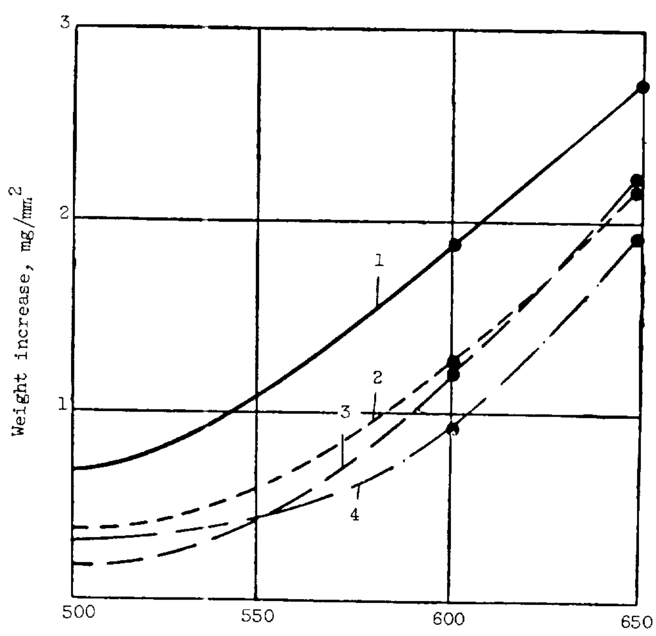

Figure 5. - Oxidation of copper and SCP at different temperatures. 1, pure copper; 2, SCP plus 1 percent MgO; 3, SCP plus 1 percent SiO_2; 4, SCP plus 1 percent Al_2O_3.